## IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant: David Harbec et al. Customer Number: 020988

Docket No.: 1770-322US Confirmation No.: 2219

 Serial No.:
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 Group Art:
 1793

 Filing Date:
 January 30, 2006
 Examiner:
 Barcena, Carlos

Title: METHOD FOR PRODUCING CARBON NANOTUBES USING A

DC NON-TRANSFERRED THERMAL PLASMA TORCH

Commissioner for Patents P.O. Box 1450 Alexandria, VA 22313-1450

# DECLARATION OF DR. JEAN-LUC MEUNIER UNDER 37 C.F.R.§ 1.132

Sir, I hereby declare and state:

- I am a joint inventor of the subject matter presently claimed in the above-identified patent application.
- 2. I received a Bachelor's Degree in Engineering Physics in 1981 from École Polytechnique Fédérale De Lausanne, Switzerland, a Masters Degree in Énergie/Physiques des Plasmas from Institut National de la Recherche Scientifique, Canada in 1983 and a Doctorate in Énergie/Physiques des Plasmas from Institut National de la Recherche Scientifique, Canada in 1986. My CV is attached to this declaration in NSERC (Natural Sciences and Engineering Research Counsel of Canada) format. I have been conducting research in the field of thermal plasma and nanomaterials for over 28 years and have authored more than 150 scientific publications on the subject.
- 3. I have reviewed the Final Office Action mailed August 11, 2009, from the US Patent & Trademark Office in respect of the above noted application, including the positions taken by the PTO with respect to several prior art references. I have also particularly reviewed the subject

matter of Smiljanic et al. (Chem. Phys. Lett., 356, 2002, 189-193), Tsantrizos et al. (US Patent No. 5395,496), (hereinafter Tsantrizos #1), Matsumoto et al. (JP07061803), Tsantrizos et al. (US Patent No. 5,147,998) (hereinafter Tsantrizos #2), Cohen et al (US Patent No. 5,993,697) and Geobegan et al. (2002/0179564).

- The present invention as claimed in the amended claims submitted simultaneously with this declaration is directed to a process for the manufacture of carbon nanostructures which may be carbon nanotubes or carbon nano-onions. The process involves the use of a high enthalpy metal electrode generated direct current thermal plasma torch which has a plasma forming gas feed and is connected to a cooled reactor. A metal catalyst is selected, which then determines the parameters of the process. Torch power may range from about 30kW up to a multi-megawatt level and the flow rate of the plasma gas feed and the reactor pressure are selected to provide a torch temperature required to vaporize and maintain the selected metal catalyst in the vapor state. A feed of a carbon containing substance and a carrier gas at a selected flow rate is provided to the cooled reactor in a quenching zone downstream of the plasma torch for the formation of carbon nanostructures. When the metal catalyst vapor is contacted by the carbon containing substance and carrier gas feed, in situ generation of metal catalyst nanoparticles having a diameter of from about 2 to about 30 nm occurs, along with the formation of atomic carbon. The metal nanoparticles act as a catalyst and nucleation points for the growth of carbon nanostructures of about the same diameter range. The carbon nanostructures are then collected from the reactor.
- 5. The selection of the metal catalyst will determine the operating parameters required for the process. Inherently, the feed of carbon containing substance and carrier gas which is fed to the cooled reactor downstream of the plasma torch into the quenching zone of carbon nanostructure formation is substantially cooler than the plasma torch flame. This allows formation of the required metal catalyst nanoparticles and the atomic carbon which form the carbon nanostructures. Keeping the catalyst vaporized until it is quenched to form the nanoparticles ultimately ensures the formation of the carbon nanostructures.
- 6. In one specific example of the present application, tungsten is selected as the catalyst metal. In my opinion, once the catalyst metal is selected, the operating parameters of the process can be determined and the plasma temperature achieved can be calculated using the parameters set for the process by the selection of the catalyst metal. An example of the calculations that one could make regarding the quench rate for the specific example in the

application is set out below. In my opinion these calculations are routine calculations that a person skilled in the relevant art could easily perform. Thus the use of the expressions "rapid quench" or "rapid cooling" have precise meaning in the context of the other parameters set out in the claims. All of this is to support the fact that the amended claims, which are all based on the description and hence are fully supported by the description, do define conditions that will provide the required rapid or fast quenching of the metal vapor that will produce the carbon nanostructures in accordance with the claimed process. The example of tungsten provided in the description together with the guidance found in the description and the claims will allow the person skilled in the art, once a catalyst metal has been selected, to determine the operating parameters that will ensure the production of carbon nanostructures every time using the claimed process.

7. Example of simplified calculations for the quench rate using data available in the patent example using He and using data given in Table I of the present application.

These calculations result in estimations of the average temperature of the plasma entering the plasma torch nozzle of around 13,000 °C, in estimations of the plasma flow velocity at the same position of around 8000 m/s (a supersonic velocity), and in estimations of the cooling rates of around 10<sup>8</sup> °C/s. Although the calculations are rough approximations of average values, these largely justify the terminology involving "high temperature", "high velocity" and "very high cooling rates".

#### Calculations:

**Torch power**: 30 – 65 kW; using **55 kW** in the present approximation, this being the power used in the demonstration experiment (US 2006/0127299A1 patent application, paragraph [0030]).

Main plasma gas flow rate: 225 slpm (paragraph [0035])

Reactor pressure: 200, 500 Torr, and stated in patent to be between 200-800 Torr He (paragraph [0034]), we use here **760 Torr** (atmospheric pressure) to simplify calculations. This has little effect on the plasma temperature inside the torch (i.e. temperature entering the nozzle). The pressure in the reactor mainly modifies the plasma properties outside of the plasma torch.

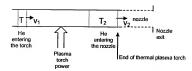
#### Mass flow rate of He:

225 slpm He =  $225x10^{-3}$  m<sup>3</sup>/min He(stp) =  $3.75x10^{-3}$  m<sup>3</sup>/s He under standard conditions

of T and p (stp).

He density (standard conditions):  $\rho_{He} = 0.178 \text{ kg/m}^3$  [1]

Mass flow rate of He:  $\dot{m} = (0.178 \text{ kg/m}^3)(3.67 \cdot 10^{-3} \text{ m}^3/\text{s}) = 6.53 \cdot 10^{-4} \text{ kg/s}$ 



Note that the carbon containing gas is injected only within the nozzle and is not present inside the plasma torch.

The change in heat energy content  $(inC_p\Delta T = inC_p(T_1 - T_1) = W_p)$  of the He gas entering at room temperature  $(T_1 = 25^{\circ}\text{C} = 298 \text{ K})$  is, in a first approximation, the result of the energy input  $W_p$  from the plasma taking the torch efficiency into consideration: Plasma torch power: 55 kW x 80% torch efficiency = 44 kW = 44 kJ/s = 44x10³ J/s Specific heat  $C_p$  of the He gas [2]:  $C_p = 5.193x10^3$  J/kg K. This value for He is a very weak function of temperature, it is for example  $5.1931x10^3$  J/kg K at 500 K, and still  $5.1931x10^3$  J/kg K at 6.200 K;  $5.210x10^3$  J/kg K at 9.000 K, and 9.200 K, and 9.200 K, at 9.000 K. In this approximation, the constant value between room temperature and

Estimated average temperature of the plasma entering the nozzle:

$$T_2 = T_1 + \frac{W_p}{\dot{m}C_p} = 298 \text{ K} + \frac{44 \cdot 10^3 \text{ J/s}}{(6.53 \cdot 10^{-4} \text{ kg/s})(5.19 \cdot 10^3 \text{ J/kg} \cdot \text{K})} = 13008 \text{ K} \cong \underline{13000 \text{ K}}$$

Note: Accurate 2-D modeling of the flow and energy fields for the plasma gas entering the nozzle yield an average temperature of the flow around 16,000 K in the present condition of 55 kW and 225 slpm He [3].

#### Estimated flow velocities:

7.500 K is used.

Using the perfect gas law linking the gas volume V with its temperature T: pV=nRT Where R is the gas constant and n the number of moles (both R and n are constant here in our two volumes at  $T_1$  and  $T_2$ ), one can evaluate the volumetric expansion of

the gas as:

$$\frac{V_2}{V_1} = \frac{T_2}{T_1} = \frac{13000 \text{ K}}{298 \text{ K}} = 43.6$$

(If one uses instead  $T_z$ =6000 K as will occur further downstream of the plasma torch and nozzle, we get:  $V_2N_1$ = 20.1)

In the above calculations we considered the pressure relatively constant inside the torch. Since the pressure at the outlet is in fact slightly smaller than the inlet pressure, the expansion ratio and the outlet velocity will be further increased.

The cross section area of the plasma torch is constant throughout the calculation domain, the inside diameter of the plasma torch tubular electrode being 5 mm, a value typical for thermal plasma torches at this power level.

Neglecting the injection velocity, a minimum value of the flow velocity  $v_1$  at the inlet can be evaluated using the volumetric flow rate of 225 slpm He =  $3.75 \times 10^{-3}$  m<sup>3</sup>/s He;

$$v_1 = \frac{\dot{V_1}}{A_1} = \frac{3.75 \cdot 10^{-3} \text{ m}^3/\text{s}}{\frac{\pi}{4} (5 \cdot 10^{-3} \text{ m})^2} = 191 \text{ m/s}$$

Similarly, the outlet volumetric flow rate  $\vec{\nu}_2$  being a factor 43.6 times higher than the inlet flow rate from thermal expansion, and the cross sectional area being constant, the flow velocity of the plasma gas entering the nozzle will be:

$$v_2 = 43.6(191 \text{ m/s}) = 8328 \cong 8330 \text{ m/s}$$

(If one uses T<sub>2</sub>=6000 K downstream of the torch and nozzle, and assuming a relatively constant jet cross section, we get: v<sub>2</sub>=3840 m/s)

Note: The more exact 2-D calculations in the model of Guo [3] leads to a velocity of roughly 5500 m/s, which is already supersonic.

#### Quench rate estimations:

The length of the nozzle is  $3.7 \text{ cm} = 3.7 \text{x} 10^{-2} \text{ m}$ . We can estimate a minimum quench rate if one assumes this is the distance travelled by the plasma gas before being cooled to 3000 K or less (Reality: this distance is in fact shorter due (a) to the presence of supersonic shocks, (b) the cold gas (200 C, paragraph [0033]) injection in the nozzle, and (c) the diffusion to the cold (in reference to the plasma) tungsten walls maintained at ~1000 C). For tungsten used in the demonstration experiments, the temperature reached for forming carbon nanotubes is its eutectic temperature around 3000 K.

Time for the gas to travel 3.7 cm : 
$$t = \frac{3.7 \cdot 10^{-2} \text{ m}}{8330 \text{ m/s}} = 4.4 \cdot 10^{-6} \text{ s}$$

(If one uses 
$$T_2$$
=6000 K we get:  $t = 9.6 \cdot 10^{-6} \text{ s}$ )

Quench rate: 
$$\frac{\Delta T}{t} = \frac{13000 \text{ K} - 3000 \text{ K}}{4.4 \cdot 10^{-6} \text{ s}} = 2.3 \cdot 10^{9} \text{ K/s}$$

(If one usesT<sub>2</sub>=6000 K we get: 
$$\frac{\Delta T}{t}$$
 = 3.1·10<sup>8</sup> K/s )

Note: The more exact 2-D modeling results of Guo [3] yields quench rates going above 5x107 K/s.

The value of 10<sup>7</sup> K/s used in the claim is conservative and strongly justifies the wording of "fast quench rates". Such value is many orders of magnitude above any quench rate values used in industrial reactors for chemical synthesis, and compares only with supersonic shock effects.

- CRC Handbook of Chemistry and Physics, 70<sup>th</sup> edition, p. B-19.
   Or available on the web at <a href="http://en.wikipedia.org/wiki/Helium">http://en.wikipedia.org/wiki/Helium</a>
- [2] Table A.1, page 393 in: M.I. Boulos, P. Fauchais, E. Pfender, <u>Thermal Plasmas Fundamentals and Applications</u>, Vol. 1, Plenum Press, New York, 1994.
- [3] L. Guo, Modeling of a supersonic DC plasma in CNT production, Ph.D. thesis, McGill University, 2009.
- 8. As the Examiner has recognized, Smiljanic et al. (Chemical Physics Letters 356, 189193, 2002; hereinafter referred to as Smiljanic), does not explicitly teach using a high enthalpy
  plasma torch with a nozzle to produce the plasma used in carbon nanotube (CNT) production.
  Smiljanic discloses the fabrication of single wall carbon nanotubes using a microwave
  plasma reactor, a system operating in the non-thermal plasma regime meaning having
  different temperatures for electrons (light species) and atoms/molecules (heavy species),
  coupled to a furnace maintained at 1300° K, and having the carbon (ethylene) and catalyst
  (vaporized ferrocene) precursors injected in a vapor form into the microwave plasma. The
  calculations set out above clearly indicate that the process conditions now specified in the
  claims will produce temperatures that are not achievable in a microwave plasma torch. A
  microwave plasma torch using argon as in Smiljanic will generate atom/molecular
  temperatures (which correspond to the "gas" temperature of importance here for vaporization
  and reaction) having a maximum value around 2000 Kelvin (see Figures 2 and 4 of ref [4], and

Section 3 and Figure 6 in ref [5] giving T<sub>g,max</sub> of 1,400 K, the authors of these papers being the inventors and suppliers of the microwave torch used by Smiljanic), and it was shown that such maximum value cannot be increased by increasing the microwave power. It is to be noted that the temperature of 5,500 K indicated in the Smiljanic paper corresponds to a nitrogen plasma and not an argon plasma as used in their experiment. A nitrogen plasma is not favorable for carbon nanotube synthesis. In comparison, a DC thermal plasma torch using nitrogen reaches temperatures over 25,000 K (see page 370 of ref [2] in the calculations above, giving measured values up to this temperature). The temperature range between ~2,000 Kelvin and ~13,000 Kelvin calculated above for the DC thermal plasma experiment is not achieveable with the microwave reactor technology of Smiljanic. This means that both the chemistry involved at these high temperatures and the treatment of refractory materials (such as tungsten used in the present application) is not possible in Smiljanic. The supersonic flow and very high cooling rate attained in the present application are also not accessible by Smiljanic. The present application specifically uses supersonic flow, tungsten metal catalyst, and high temperature chemistry (atomic carbon generated from C<sub>2</sub>C<sub>14</sub> molecule and reactions at temperatures above CI-based side reactions) in the claimed process to generate the carbon nanotubes which are not found in Smiljanic.

- [4] H. Nowakowska, Z. Zakrzewski, M. Moisan, and M. Lubanski, Propagation characteristics of surface waves sustaining atmospheric pressure discharges: the influence of the discharge processes, J. Phys. D: Appl. Phys. 31, 1422-1432, 1998.
- [5] M.D. Calzada, M. Moisan, A. Gamero, A. Sola, Experimental investigation and characterization of the departure from local thermodynamic equilibrium along a surface-wave-sustained discharge at atmospheric pressure, J. Appl. Phys., 80, 1, 1996.
- 9. In Tsantrizos et al (USPN 5,395,496, hereafter Tsantrizos #1), there is disclosed the use of a DC thermal plasma torch for the **homogeneous chemical reaction process** to form fullerene molecules ( $C_{60}$  and  $C_{70}$ ) in a plasma environment specifically attempting to maintain a large and uniform temperature environment. Thus a combination of Tsantrizos #1 and Smiljanic does not provide the presently claimed process, since the vaporization of the catalyst metal to provide metal catalyst nanoparticles which act as nucleation points and catalyst for the growth of carbon nanostructures is not arrived at. There is no teaching of the handling of a metal vapor carrying thermal plasma jet for a heterogeneous reaction process based on the nanoparticles nucleated from the metal vapors.
- In Matsumoto et al. (JP 07061803), there is disclosed the use of an inductively coupled plasma using carbon powders only, with no use of catalyst being specified, to form a very

low yield (5-10%) of a mixture of fullerenes and carbon nanotubes. Matsumoto does not teach a heterogeneous reaction involving the handling of metal vapors for the formation of nanoparticles of catalyst. This reference does not motivate the combination of Smiljanic and Tsantrizos #1 for these reasons.

- 11. In my opinion therefore, it is clear that combining Smiljanic and Tsantrizos #1, with the teachings of Matsumoto in mind, does not lead to the presently claimed process which involves rapid quench rates and conditions leading to supersonic flows, such as jet expansion in the nozzle, pressures below atmospheric, and very high plasma flow rates, which are not taught or suggested in the combination of the three references.
- 12. In Geobegan et al (US2002/0179564A1; thereafter called Geobegan), a method is established for the generation of carbon nanorods and carbon nanotube structures in a two-step process termed "condensed phase conversion growth" (paragraph [0057] and claims 4, 8, 9 in Geobegan) in which a first step is providing a "condensed phase" matrix material deposited on a surface (Claims, 4, 9 in Geobegan), which condensed phase does not contain the carbon nanotube material but rather contains mixtures of carbon powder and catalyst powder in an aggregated state (Figure 1 in Geobegan). This is followed by a second post-treatment step called the "conversion growth" of the nanotube structure by a treatment of the "solid or powdered material instead of vapor" (paragraph [0056] in Geobegan), which solid/powder material was deposited in the first step. In their proof of principle experiments, the deposit formed in the first step is later heated in vacuum or in a gas to initiate the growth of carbon nanotubes. Various strategies, including DC thermal plasma torch, are used to produce the initial condensed phase acting as a source material (paragraph [0059] in Geobegan). These strategies highlight the importance of supplying the particles to a substrate at a high rate in a particle conversion epitaxy process (paragraph [0062] in Geobegan), i.e for the formation of the solid precursor deposit. The method in Geobegan describes a transformation to the carbon nano-structures after the deposition of a solid precursor in a post-processing step in a way similar to the chemical vapor deposition (CVD) processes for the growth of carbon nanotubes on surfaces and structural components (paragraph [0090] in Geobegan). Geobegan does not teach a method to generate carbon nanotubes in the gas phase. Geobegan also does not teach a control of the gas dynamics that would make possible the formation of carbon nanotubes in flight within a thermal plasma jet making a continuous one-step formation process possible as in the present application. Geobegan teaches a method to generate a solid precursor material

that makes possible the CVD-like carbon nanotube process to be made in a postprocessing step.

- 13. I am also of the opinion that back in 2002, it was not known by those skilled in the art or from the literature that there could be advantages to the use of rapid quench rates for example from a supersonic flow, as both systems of Smiljanic and Tsantrizos teach a requirement of relatively uniform conditions; Smiljanic uses an oven at the outlet of his microwave plasma to maintain longer residence times at a uniform temperature, while Tsantrizos sets a spherical geometry of the reactor in order to generate more uniform temperature profiles. Even at the present time, other researchers and experts in the field of thermal plasmas are indicating their unsuccessful attempts to produce CNTs using the DC thermal plasma torch technology (this was officially indicated at the 2007 Round Table on Thermal Plasma Technology, Sharm El Sheik, Egypt; it also follows from the absence of thermal plasma torch papers on this topic (apart from those of Harbec and Meunier) in the International Symposium on Plasma Chemistry (most important international conference for thermal plasma technology) up to year 2007).
- 14. Copies of all references referred to herein are attached to this Declaration.
- 15. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true, and further, that these statements were made with the knowledge that willful false statements and the like, so made, are punishable by fine or imprisonment, or both, under 18 U.S.C. §1001 and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Respectfully submitted

November 7 2009

Dr. Jean-Luc Meunier

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Degree	Name	of discipline	Institution			Country			Date yyyy/mm
Bachelor's	Engineering Physics		École Polytechnique Fédérale de Lausanne		de	SWITZERLAND		1981/01	
Master's	Énergie / Physique des		Institut national de recherche scientifique-Énergie			CANADA		1983/04	
Doctorate	Énergie /	Physique des	Institut national de recherche			CANADA			1986/07
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		Supervised	Co-supervised	Supervis	sed	Co-supervis	sed	Т	otal
Undergraduate		5	1	10			16		16
Master's		1		5		6	12		12
Doctoral		1	3	2			6		6
Postdoctoral		1		3				4	
Others				1					1
Total		8	4	21		6		39	

Personal identification no. (PIN) Family name

17858 Meunier

ACADEMIC, RESEARCH AND INDUSTRIAL EXPERIENCE (use one additional page if necessary) Period (yyyy/mm Position held (begin with current) Organization Department to yyyy/mm) Associate Professor McGill Chemical Engineering 1996/06 Assistant Professor McGill University Chemical Engineering 1990/06 to 1996/05 Research Associate McGill University Chemical Engineering 1986/08 to 1990/05 Research Engineer Hydro-Ouebec Institut de recherche 1984/08 H.-Q. (IREQ) to 1986/08 Research Assistant École Polytechnique Fédérale de C. Recher en physique 1980/07 Lausanne des plasmas (CRPP) to 1981/06 Research Assistant École Polytechnique Fédérale de IMAC, Génie Civil 1978/06 Lausanne to 1978/09



Personal identification no. (PIN) Family name

per year

17858 Meunier RESEARCH SUPPORT Years of Title of proposal, funding source and program, and time commitment (hours/month) Family name and initial(s) Amount tenure

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List all sources of support (including NS past four (4) years but now completed; to funding directly applicable to your research.	ERC grants and university start-up funds) held as an applicant or a ) support currently held, and c) support applied for. For group grants, i ch. Use additional pages as required.	co-applicant: a)	support hentage of	eld in the he
a) Support held in the past 4 y	ears			
Terreault B., 23 univ. professors, 16 ind. res.	Plasma Québec - Réseau thématique en sciences et applications avancées des plasmas FCAR Regroupements stratégiques 5 hours/month	440,000 440,000 440,000 440,000	(5%) (5%)	2003 2004
Meunier, J-L (P.I.), Coulombe, Munz, Gauvin, Drew	Development of new advanced electrode materials for electric arc devices CRSNG Strategic projects	202,720 173,720 173,720	(35%)	2004
Coulombe, S (P.I.), Meunier JL, Gauvin R	Développement d'un procédé plasma pour la synthèse des nanofluides FQRNT Équipe 10 hours/month	51,250 51,250 43,750	(20%)	2006 2007 2008
Margot, J. (PI) + 39 researchers from 4 Universiti	Plasma-Québec: Un regroupement stratégique en sciences et applications des plasmas FQRNT Centre 10 hours/month	140,000 140,000	(3%) (3%)	2006 2007

of applicant

Personal identification no. (PIN) Family name

17858 Meunier
RESEARCH SUPPORT

oars of Family name and initial(s) Title of proposal, funding source and program, Amount tenure of applicant and time commitment (hours/month) per year (yyyy) List all sources of support (including NSERC grants and university start-up funds) held as an applicant or a co-applicant: a) support held in the past four (4) years but now completed; b) support currently held, and c) support applied for. For group grants, indicate the percentage of the funding directly applicable to your research. Use additional pages as required. a) Support held in the past 4 years Meunier, J.-L. Modélisation numérique d'une torche à plasma 15,000 2008 DC à air soufflé dans un réacteur de gazéfication FORNT/MITACS Programme de stage post-doctoral 10 hours/month Meunier, J.-L. Modélisation numérique d'une torche à plasma 15,000 2008 DC à air soufflé dans un réacteur de gazéfication PyroGenesis Inc. Programme de stage post-doctoral FORNT/MITACS (contr. ind.) 10 hours/month b) Support currently held Meunier, Jean-Luc Plasma-surface interactions for nano-comp. 32.500 2005 materials and arc-electrode phen. 32,500 2006 NSERC 32.500 2007 Discovery Grant 32,500 2008 32,500 2009 35 hours/month Stansfield, B(P.I.). The synthesis and functionalization of 146,200 (35%) 2006 Dodelet, Berk, Meunier nanostructured carbon black by plasma for use in 136,200 (35%) 2007 fuel cells 144,700 (35%) 2008 NSERC Strategic 10 hours/month

Personal identification no. (PIN) Family name

Meunier

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RESEARCH SUPPORT

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List all sources of support (including NSERC grants and university start-up funds) held as an applicant or a co-applicant: a) support held in the

List all sources of support (including NSERC grants and university start-up funds) held as an applicant or a co-applicant: a) support held in the past four (4) years but now completed; b) support currently held, and c) support applied for. For group grants, indicate the percentage of the funding directly applicable to your research. Use additional pages as required.

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b) Support currently held	λ 11			
Chaker M. (PI) & Plasma-Québec researchers	Laboratoire en sciences et applications des plasmas Fondation canadienne pour l'innovation Fonds d'opération des infrastructures (McGill share only)	27,551 32,995 32,995 32,995 32,995	(33%) (33%) (33%)	2008 2009 2010
Meunier JL., Coulombe S., Munz R.J.	Plasma-Québec/McGill McGill University VP(Research) Centers	14,037 14,037		
Margot J, and Plasma-Qu/bec members	Regroupement Plasma-Québec FQRNT Regroupements stratégiques	330,000 330,000 330,000 330,000 330,000	(5%) (5%) (5%) (5%) (5%)	2008 2009 2010 2011 2012
Meunier JL.	Modélisation numérique de torches à plasma DC à air soufflé - 2e partie FQRNT/NSERC/Mitacs Programme de stage post-doctoral 10 hours/month	20,000		2009

Personal identification no. (PIN) Family name

17858 Meunier

Family name and initial(s) of applicant	Title of proposal, funding source and program, and time commitment (hours/month)	Amount per year	Years of tenure (yyyy)
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Meunier JL.	Modélisation numérique de torches à plasma DC à air soufflé - 2e partie PyroGenesis Inc. Programme de stage post-doctoral (contr. ind.) 10 hours/month	10,000	2009
c) Support applied for			
Coulombe, Sylvain (PI); Meunier JL	Mercury-free discharge lamp based on CNT array electrodes NSERC Idea to Innovation, Phase 1 10 hours/month	108,750 (50%)	2010
Coulombe, Sylvain (PI) + 8 researchers	Tabletop scanning electron microscope NSERC Research Tools and Infrastructure	118,576 (12%)	2010
Omanovic, Sasha	Imaging Ellipsometry System for Advanced Materials Characterization NSERC Research Tools and Instrumentation - Category 1	129,554 (5%)	2010

# Highly Qualified Personnel (HQP)

Provide personal data about the HQP that you currently, or over the past six years, have supervised or co-supervised.

			Personal identification no. (PIN)	amily name	
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Name Type of HQP Training and Status		Years Supervised or Co-supervised	Title of Project or Thesis	Present Position	
Vandsburger, Leron	Doctoral (In Progress)	Co-supervised 2009 -	Study of carbon nanotubes based electrodes for fluorescent	Ph.D. student, McGill University	
Mendoza Gonzalez,	Postdoctoral (In Progress)	Supervised 2008 -	Numerical Modeling of a DC Thermal Plasma Torch	PDF, Chemical Engineering, McGill University	
Pristavita-Tur Ramona	Doctoral (In Progress)	Co-supervised 2007 -	Functionalized carbon production for PEM fuel cells	Ph.D. student, McGill University	
Baddour, Carole	Doctoral (In Progress)	Supervised 2005 -	CNT/TiN nano-composite films of stainless steel	Ph.D. student, McGill University	
Dionne, Martin	Doctoral (In Progress)	Co-supervised 2005 -	Modeling and exp. study of plasma/CNT surface interaction	Ph.D. student, McGill University	
Larissa, Jorge	Undergraduate (Completed)	Co-supervised 2009 - 2009	Polymer-CNT composites using plasma functionalization	UG student, McGill University	
Morier, Geoffrey	Undergraduate (Completed)	Supervised 2009 - 2009	DLC films using an arc-PVD source	UG student, McGill University	
Pascone, Pierre-Alexan	Undergraduate (Completed)	Supervised 2009 - 2009	Electrochemical surface area measurements of CNT covered el	UG student, McGill University	
Pasieka, James J.	Undergraduate (Completed)	Supervised 2009 - 2009	CNT ablation study using a CO2 laser	UG student, McGill University	
Upham, David	Undergraduate (Completed)	Supervised 2009 - 2009	Th-CVD of CNT in a fluidized be (Part 2)	ed UG student, McGill University	
Valla, Sebastien	Undergraduate (Completed)	Supervised 2009 - 2009	CFD modeling of the flow/temperature fields in CVD	UG student, McGill University	
Vandsburger, Leron	Master (Thesis) (Completed)	Co-supervised 2007 - 2009	Synthesis & covalent surface modification of carbon nano	Ph.D. student. McGill University	
Guo, Liping	Postdoctoral (Completed)	Supervised 2008 - 2008	Modeling of a DC thermal plasmatorch	Shaw Energy & Chemical Group, USA	
Jaung, Sarah	Undergraduate (Completed)	Supervised 2008 - 2008	Characterization of SS substrate surface prior to CNT growth	UG student, McGill University	
Moran, Blain	Undergraduate (Completed)	Supervised 2008 - 2008	Study of PAH content in carbon black in the context of fuel	UG student, McGill University	
Jpham, David	Undergraduate (Completed)	Supervised 2008 - 2008	Th-CVD of CNT on SS powders (part 1)	UG student, McGill University	
Azem, Amir	Master(Thesis) (Completed)	Supervised 2005 - 2008	Growth of nitrogen doped diamon films using ICP	(Germany)	
Guo, Liping	Doctoral (Completed)	Supervised 2002 - 2008	Modeling of a supersonic DC plasma torch in CNT production	Shaw Energy & Chemical Group, USA	
Aitra, Reema	Undergraduate (Completed)	Supervised 2007 - 2007	Th-CVD growth of CNT on SS: surface morphology opt.	(unknown - recently graduated)	
			Synthesis of CNT applications in electreoch. capacitors	Ph.D. student, McGill University	

Form 100 (2009 W), page 4 of 4

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# Highly Qualified Personnel (HQP)

Provide personal data about the HQP that you currently, or over the past six years, have supervised or co-supervised.

			Personal identification no. (PIN)	Family name
			17858	Meunier
Name	Type of HQP Training and Status	Years Supervised or Co-supervised	Title of Project or Thesis	Present Position
Dai, Gui-Ping	Postdoctoral (Completed)	Supervised 2005 - 2007	CNT synthesis using a thermal plasma process	Research Associate, USA
Fadlallah, Faysal	Master(Thesis) (Completed)	Supervised 2005 - 2007	Prep. of metal-CNT composite powders for th. plasma spraying	Future Pipe Industries, Dubai
Reddy, Naveen K.	Master(Thesis) (Completed)		CVD growth of oriented carbon nanotubes for electrode applic	Ph.D. student, Belgium
ElMallah, Nadine	Master(Thesis) (Completed)	Supervised 2004 - 2006	CNT transport in a PVD reactor for arc electrode application	Industry, Canada
Hinkov, Ivaylo	Postdoctoral (Completed)	Supervised 2004 - 2006	CNT synthesis using inductivel coupled plasma with C2Cl4 pc	y Researcher, France
Roy, François	Master(Thesis) (Completed)	Co-supervised 2004 - 2006	Nanoscale island of dielectric material on Cu by ion implant	Ph.D., EPFL, Switzerland
Harbec, David	Doctoral (Completed)	Supervised 2002 - 2006	Carbon nanotube catalytic synthesis using dc plasma torch	Post-doc, Université de Sherbrooke
Beaulieu-Berg S.	Undergraduate (Completed)	Supervised 2004 - 2004	Plasma spraying of Cu-CNT nanocomposite	PhD Student, McGill University
(Name withheld)	Undergraduate (Completed)	Supervised 2004 - 2004	Modeling of the temperature profile in graphite are electrod	(unknown)
(Name withheld)	Undergraduate (Completed)	Supervised 2004 - 2004	Modeling heat transfer in graph arc electrode for CNT syn	ite (unknown)
(Name withheld)	Undergraduate (Completed)	Supervised 2004 - 2004	CNT synthesis from arc-based device in liquid precursor	(unknown)
(Name withheld)	Master(non-th.) (Completed)	Supervised 2004 - 2004	Study of fullerene content from CNT reactor based on C2Cl4	unknown
	Master(Thesis) (Completed)		Nanocomposite formation based oin C60 imbedded in polymer fi	
	Undergraduate (Completed)		TGA calibration for CNT characterization from synth.	(unknown)
Mélanie	Undergraduate (Completed)		Raman spectroscopy on CNT containing soot	(unknown)

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# Part II. Research Contributions (updated October 24 2009)

## 1. Most Significant Contributions to Research and Applications

#### 1.1 Arc/cathode surface interaction: Erosion and AIP contributions

Success in modeling the local plasma-cathode phenomena led to important scientific advances in the understanding of this interaction, with theoretical and experimental expertise developed over the last 20 years and leading to three (3) patents (2 of these applied in 2009) and 8 journal papers in the last 6 years; we are forming one of the leading team in the world in this area. A global approach to solve problems of electrode degradation using nano-composite materials is presently leading to a new family of electrode systems based on carbon nanotubes; the two patent applications made in 2009 are based on (1) a new low voltage high current flux cathode source from a "properly" designed nanotube array, and (2) from an innovative open nanocomposite "felt" structure yielding 3D volumetric emission from the surface.

## 1.2 Carbon nanotubes synthesis and applications

We first developed a process for the production of carbon nanotubes (CNT) and fullerenes using a supersonic thermal plasma technology (Patent WO-A1 2004046030) and a liquid or gas carbon precursor. The process is developed using a 100kW DC plasma torch, and has an important scale-up potential for industrial production of this advanced material (on this topic in the last 6 years: 4 graduate student projects, 2 USRA, 2 journal papers, 10 conference papers). We also developed recently the technology to grow carbon nanotubes directly on stainless steel metal surfaces without the need of an external catalyst, a technique that is now opening a wide range of applications and showing an important impact on the research community, as reflected by the 6 graduate students and 8 USRA projects on this topic in the last 6 years (+2 journal & 9 conf. papers). Both techniques discussed above generated papers in the journal Carbon, a highly rated international journal (impact factor: 4.37). Another technique developed recently relates to an "easy" template formation and nanotube growth process on aluminum oxide (see the first 2009 patent), this technique providing the surface morphology for electrode with strongly enhanced electron emission (3 journal & 9 conf. papers, including the best paper award at ISPC-19(2009) in Germany [99]). The CNT work was generic and now applies to novel devices (new plasma source by M. Dionne, Ph.D) and original concepts (3-D open volumes on surfaces, C. Baddour, Ph.D). Other applications in the fields of energy and materials are now studied. In parallel to CNT work and using the expertise generated in this field, new carbon based materials (carbon nano-flakes) are now being developed for advanced catalyst structures and platinum replacement in fuel cells.

#### 1.3 Diamond films and RF thermal plasma CVD

Research on diamond film growth using bias assisted thermal plasmas provided local values in the diamond growth process. This was a very challenging task as the substrates are immersed in thermal plasma flames (~5000K) with RF floating potentials (~1 kV) oscillating at 4 MHz. Such local values gave in situ real time indications on the film nucleation and growth processes to a point where new monitoring and control parameters have been defined. These results led to the growth of nitrogen doped diamond films for potential applications in electrochemical devices (ex.: for water treatment).

# 2. Research Contributions (Bold: graduate/UG students supervised)

### 2.1 Refereed journal publications: (2003-present)

- M. Dionne, S. Coulombe, J.-L. Meunier, Energy exchange during electron emission from carbon nanotubes: Considerations on tip cooling effect and destruction of the emitter, Phys. Rev. B, 80, 085429, 2009.
- L. Vandsburger, E.J. Swanson, J. Tavares, J.-L. Meunier, S. Coulombe, Stabilized aqueous dispersion of multi-walled carbon nanotubes obtained by RF glow discharge treatment, J. Nanoparticle Research, 11, 7, 1817-22 (DOI 10.1007/s11051-009-9656-4) 2009.
- C.E. Baddour, F. Fadlallah, D. Nasuhoglu, R. Mitra, L. Vandsburger, J.-L. Meunier, A simple thermal CVD method for carbon nanotube synthesis on stainless steel 304 without the addition of an external catalyst, CARBON, Vol. 47, 1, pp. 313-318, (doi:10.1016/j.carbon.2008.10.038) 2009.
- J. Tavares, S. Coulombe, J.-L. Meunier, Synthesis of cubic-structured monocrystalline titanium nitride nanoparticles by means of a dual plasma process, J. Phys. D: Appl. Phys, 42, 10, 1-4, 2009.
- M. Kandah, J.-L. Meunier, Production of Carbon Nanotubes-Nickel Composites on Different Graphite Substrates, Fluid Dynamics and Materials Processing, Vol. 5, No. 2, 123-136, 2009.
- M. Dionne, S. Coulombe, J.-L. Meunier, Field emission calculations revisited with Murphy & Good theory: a new interpretation of the Fowler-Nordheim plot, J. Phys. D: Appl. Phys., 41,24,1-8, 2008.
- M. Kandah, J.-L. Meunier, Production of carbon nanotubes on different monel substrates, Fluid Dynamics & Materials Processing, Vol. 4, No. 4, pp. 231-236, 2008.
- 44. M. Dionne, S. Coulombe, J.-L. Meunier, Screening effects between field-enhancing patterned carbon nanotubes: A numerical study, IEEE Trans. Electron Dev., Vol. 55, 6, 1298-1305, 2008.
- L. Rao, R.J. Munz, J.-L. Meunier, Vacuum arc velocity and erosion rate measurements on nanostructured plasma and HVOF coatings, J. Phys. D: Appl. Phys. 40, 4192-4201, 2007.
- 42. D. Harbec, J.-L. Meunier, L. Guo, J. Jureidini, A parametric study of carbon nanotubes production from tetrachloroethylene using a supersonic thermal plasma jet, Carbon, 45, 2054-2064, 2007.
- F. Roy, G. Abel, B. Terreault, A. Reguer, J.-L. Meunier, M. Bolduc and G.G. Ross, On-line system for temperature and accumulated dose control in Plasma-Based Ion Implantation, Rev. Sci. Instrum., 78, 023905, (doi: 10.1063/1.2472601) 2007.
- J.-L. Meunier, S. Coulombe, M. Kandah, Erosion of carbon arc cathodes operating in the thermofield electron emission mode, Plasma Sources Sci. & Technol., 16, 33-41, 2007.
- M. Kandah, J.-L. Meunier, Removal of Nickel Ions from Water by Multi-Walled Carbon Nanotubes, J. Hazardous Materials, 146, 1-2, 283-288, July 2007.
- L. Rao, N.K. Reddy, S. Coulombe, J.-L. Meunier, R. J. Munz, Carbon Nanotubes as Nanoparticles Collector, Journal of Nanoparticle Research, 9, 4 689-695, 2007.
- L. Rao, R.J. Munz, J.-L. Meunier, Vacuum arc velocity and erosion rate measurements on nanostructured plasma and HVOF spray coatings, J. Phys. D: Appl. Phys. 41, 19,4192-4201, 2008.
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- N.K. Reddy, J.-L. Meunier, S. Coulombe, Growth of carbon nanotubes directly on a nickel surface by thermal CVD, Materials Letters, 60, 3761-3765, 2006.
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# 2.2 Conference Contributions : (2003-present) (\* = refereed with proceedings)

- 102.\* Pristavita R., Meunier J.-L. and Berk D., Extraction and identification of volatile compounds from carbon black produced by thermal plasma, 8<sup>th</sup> World Congress in Chemical Engineering, Montreal (Canada), Aug 23-27 2009.
- 101.\* Tavares J., Coulombe S. and Meunier J.-L., Dual plasma synthesis of single-crystal titanium nitride nanoparticles, ICPIG-29, Cancun (Mexico), July 12-17 2009.
- 100.\* Baddour C., Meunier J.-L., Carbon nanotube synthesis on stainless steel 304 by thermal CVD without the addition of an external catalyst: surface characterization, Carbon'09, Biarritz (France), June 2009.
- 99.\* (<u>Best paper award</u>) Dionne M., Coulombe S., Meunier J.-L., *Plasma-Enhanced Electron Emission from Carbon Nanotube Array Cathodes*, Paper 1.1.9, 19<sup>th</sup> Intl. Symp. on Plasma Chemistry, Bochum, Germany, July 2009.
- 98.\* Pristavita R., Meunier J.-L., Berk D., The Synthesis and Functionalization of Nanostructured Carbon Black by Thermal Plasma for Use in PEM Fuel Cells, Paper P3.16.1, 19<sup>th</sup> Intl. Symp. on Plasma Chemistry, Bochum, Germany, July 2009.
- 97.\* Mendoza Gonzalez N., Lakshminarayana R., Carabin P., Meunier J.-L., A Three Dimensional Model of a DC Plasma Torch Used in Waste Treatment Applications, Paper 2.2.24, 19th Intl. Symp. on Plasma Chemistry, Bochum, Germany, July 2009.
- 96.\* Azem. A, Meunier J.-L., Nitrogen Doped Diamond TP-CVD Deposition by a RF-Inductively Coupled Plasma, Paper 3.7.1, 19th Intl. Symp. on Plasma Chemistry, Bochum, Germany, July 2009.

- 95.\* Vandsburger L., Tavares J., Coulombe S., and Meunier J.-L., Surface modification of multi-walled carbon nanotubes for enhanced dispersion and stability using a RF glow discharge, 19th Intl. Symp. on Plasma Chemistry. Bochum. Germany. July 2009.
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- Mendoza-Gonzalez N.Y., Pristavita R., Meunier J.-L., Berk D., Synthèse de noir de carbone fonctionalisé
  par plasma thermique ICP: Optimisation numérique, Colloque Plasma-Québec, Montréal, May 2009.
- Vandsburger L., Coulombe S. et Meunier J. -L., "Projet d'incorporation de nanotubes de carbone dans un nanofluide: Problématique et méthodologie," Plasma-Québec meeting, 22-23 mai 2008
- Pristavita R., Meunier J.-L., Berk D., Synthèse et fonctionalisation de noir de carbone nanostructuré par plasma thermique pour les piles à combustible PEM, Plasma-Ouébec meeting, 22-23 mai 2008.
- 87.\* Dionne M., Coulombe S. and Meunier J.-L., Energy exchange during electron emission from carbon nanotubes: from the tip cooling effect to the destruction of the emitter, COMSOL Conference, Boston (USA), October 9-11 2008.
- 86. Dionne M., Meunier J.-L. and Coulombe S., Enhancing electron emission from surfaces: carbon nanotube arrays on commercial grade anodized aluminum, Functional Coatings and Surface Engineering Symposium, Montréal (QC), June 1-4 2008.
- Dionne M., Meunier J.-L., Coulombe S., Optimisation de l'émission électronique par des réseaux de nanotubes de carbone insérés dans l'aluminium commercial anodisé, Plasma-Québec meeting, 22-23 mai 2008.
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- 74.\* NK. Reddy, L. Rao, C. Qin, S. Coulombe, J.-L. Meunier, R.J. Munz, Carbon nanotubes as filters for nanoparticles, 56<sup>th</sup> Canadian Chemical Engineering Conf., U. de Sherbrooke, Oct. 15-18, 2006.
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#### 2.3 Invited talks:

- J.-L. Meunier (<u>Invited Paper</u>), D. Harbec, L. Guo, Thermal Plasma Synthesis: Processes Involving Both the Catalyst Precursor Generation and the Nano-structures Growth on this Catalyst, Intl Round Table on Thermal Plasma Technology, Sharm El Sheik, Jan 2007.
- J.-L. Meunier, invited paper, joint CAP Congress/Photonics North Congress, Québec, June 3 2002 (Ref. 56 in Conference section 2.2 above)
- J.-L. Meunier, invited lecture, Club EDF-Arcs Électrique Meeting, Paris, France, March 15, 2000 (Ref. 47 in Conference section 2.2 above).
- J.-L. Meunier, Conférencier invité, Intéraction arc-cathode froide, INSA (Institut National de Sciences Appliquées), Toulouse, France, 5 avril 2000.
- J.-L. Meunier, Conférencier invité, Intéraction arc-électrode, LAEPT, Université Blaise Pascal, Clermont-Ferrand, France, 13 avril 2000.
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  J.-M. Meunier, invited lecture, Arc-Cold Cathode Interaction, Chemical Engineering Department, A&M University, College Station, Texas, USA, May 10 2000.
- J.-L. Meunier, conférencier invité, Intraction plasma d'arc électrique cathode froide: Propriétés et importance d'une pression locale élevée au voisinage du spot, INRS-Énergie et Matériaux, Varennes, Qc, Canada, 16 juin 2000.
- J.-L. Meunier, invited talk, Cold cathode arc attachment: the importance of the high local pressure, 13<sup>th</sup> Int. Symp. on Plasma Chem.(ISPC-13), Beijing, China, Aug.1997 (ref. 36 in section 2.2 above).
- J.-L. Meunier, invited talk, Fullerene studies from cathodic arc carbon plasma expansion in helium, Department of Chemistry, Warsaw University, Poland, 1994.
- J.-L. Meunier, invited lecture, Cathode spot phenomena, Laboratoire de décharges dans les gaz, CPAT, Université Paul Sabatier, Toulouse, France, 1994.

#### 2.4 Patents

- C. Baddour and J.-L. Meunier, Simple method for the production of carbon nanotube composites and the novel nanocomposites produced thereof, US PTO application No. 61183620, June 3 2009.
- M. Dionne, S. Coulombe and J.-L. Meunier, Anodic aluminum oxide templates with 2-D linear ordering on commercial grade aluminum, US PTO application, May 2009.
- D. Harbec and J.-L. Meunier, Method for producing carbon nanotubes using a thermal plasma torch, Publication number: 2004046030/WO-A1; date: June 3, 2004.

 J.-L. Meunier and M. Kandah, Particle-free cathodic arc carbon ion source, US patent Number: US 6,261,421 B1; July 17 2001.

## 3. Other Evidence of Impact and Contributions

- Paper [99] (Germany, 2009) in section 2.2 received the "best paper award" in this international conference. ISPC (Intl. Symp. on Plasma Chemistry) has historically been the most important conference in our field, and this award was attributed, on top of the quality of the paper, to the significance of the work enabling new plasma devices with optimal surface emission processes, i.e. to our "new family of electrodes".
- 2. The conference paper [75] (Sect. 2.2) was on invitation only, and was aimed at grouping world leaders in the thermal plasma community in order to assess the state of the technology and try to assess needs and orientations we should target in the years to come. My invitation to give a talk related to the new innovative trend we developed by being able to first nucleate homogeneously nano-scale templates in the thermal plasma stream, and on this template grow heterogeneously the nanoscale product of interest (CNT in our case) downstream of the same plasma stream. This concept was entirely new.
- 3. The talk given by Baddour in Biaritz (Sect. 2.2 ref [100]) followed our paper in the journal Carbon (Sect 2.1 ref [49]). This paper attracted attendees from the other 3 parallel sessions and literally crowded the conference room for the duration of the talk, indicating the large interest generated by the new developments.

# 5. Contributions to the training of HQP

The HQP list in Part I of this NSERC CV covers the years 2003-2009. A total of over 30 graduate students have been formed since the late 1980s.

The number of graduate students generally goes with the funding, the interest of UG students however is a good sign of the passion one team may develop, particularly when these UG students follow to M.Eng. degrees within the team. A total of 16 UG students were formed in the last 6 years, and 3 (possibly 4) have or are intending to continue towards a M.Eng.(Thesis). I tend to give USRA projects with formation on state of the art technologies and research topics in line with our current needs of understanding and applications, i.e. publishable results.